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ORIGINAL ARTICLE

Determination of the moisture content in wood chips of Scots pine and Norway spruce using Mantex Desktop Scanner based on dual energy X-ray absorptiometry

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Abstract There has been a longstanding interest in developing a fast and accurate method for measuring moisture content in wood. The gravimetric method that is commonly used today is a widely accepted industrial standard but it is time consuming. In this study, we tested and evaluated Mantex Desktop Scanner that is based on dual energy X-ray absorptiometry as a potential technique for fast and accurate determination of moisture content at different temperatures in wood chips of *Pinus sylvestris*, *Picea abies* and a variety of mixtures thereof. The results of the study give similar results as the gravimetric method when determining the moisture content in wood. They further show that there was no significant influence on the result if samples were measured while frozen or at room temperature.

Keywords Dry matter content · Moisture content · *Picea abies* · *Pinus sylvestris* · Wood chips

Introduction

The supply of wood chips from sawmills to pulp and paper mills in Sweden contributes with approximately 25 % or 11 million m^3 solid under bark of the overall wood use in the industry [1]. The entire quantity of sawmill chips to the pulp and paper mills in the Nordic countries consists of Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies*). The sawmill chips are normally purchased according to dry weight. The traditional way to determine the dry weight of wood chips is to weigh a sample of chips, dry them in an oven, and weigh them again after they have reached a constant weight. In this manner, the moisture content (MC) can be calculated to determine the dry weight for the entire load of wood chips (gravimetric method). This procedure is very tedious because it is necessary to wait for at least 24 h until the wood chips reach a constant weight. However, this procedure is common practice today at all Swedish pulpmills. The relatively long-time span it takes for the samples to dry makes it harder to optimize the flow of wood chips that arrive at the mill. For example, sorting loads of chips with similar MC into different stacks is not possible today, even though that would be of interest for the pulpmill since the MC is an important process parameter when producing pulp [2–5].

Several studies have attempted to develop and evaluate other methods, such as radio frequency and near infrared, that could quickly and accurately determine the MC of wood chips; none of them have been truly successful [6–11]. However, Hultnäs et al. [12] performed a study with Mantex Desktop Scanner (MDS) that used dual energy X-ray absorptiometry on wood chips of Norway spruce with a calibration model particularly developed for spruce, that showed promising results for that particular species, with good coherence with the gravimetric method that was used as a reference. Scots pine and Norway spruce contribute with 80 %, about 40 %, respectively, to the woodstock in Sweden [13]. It is therefore of importance that the dual energy X-ray absorptiometry technique can be used for both species. The dual energy X-ray absorptiometry technique uses the fact that every material has a unique absorption of X-ray energy; this feature can be used to measure a specific substance in a material, e.g. water in wood. Even though pine and spruce are softwoods and very

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similar in their chemical composition, there are some small differences [14]. These differences could influence the result of the MC measurement in a negative way, if using a calibration model developed for spruce when determining the MC in pine or vice versa. In addition, several other methods used to determine the MC in wood have showed difficulties when it comes to determine the MC in frozen or semi frozen material [15]. This is a problem in the Nordic region where frozen wood arriving at the pulpmills can occur during up to 6 months per year.

The aim of the research presented in this article is to determine whether it is possible to use the same calibration model for the MDS that was developed for spruce [12] to determine the MC in pine wood chips with the same level of accuracy and precision compared to the gravimetric method. Second, if a calibration model is developed only for pine, would it produce results with better precision and accuracy as compared to the use of a calibration model developed for spruce when measuring the MC in pine? Furthermore, is it possible to use a single calibration model that can determine the MC in both pine, spruce and different mixtures thereof with good compatibility compared to the results of the gravimetric method? Finally, are there any significant differences in the results when measuring the MC with the MDS on frozen and unfrozen samples?

The first part of this study was performed as a bachelor thesis by Fernandez-Cano [16].

Technique description

The application tested in this study is based on dual energy X-ray absorptiometry and is developed to measure the MC in organic material, mainly wood. The MDS X-rays the sample of wood chips with two different energies, 90 kVp, 2 mA and 40 kVp, 5 mA; this gives effective energies of approximately 58 and 30 keV. The MDS consists of an X-ray tube and a semiconductor linear array detector that is placed on the opposite side of the X-ray tube. A schematic setup of the MDS is found in Fig. 1. The sample of wood chips is placed in a plastic vessel of approximately 3 l and

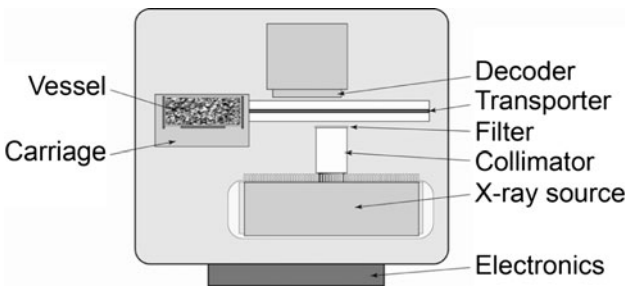

Fig. 1 A schematic setup of the MDS

is automatically driven on a carriage between the X-ray source and its collimator and the decoder twice per measurement. The first time it is scanned with the lower energy and the second time with the higher energy [17].

The theory of the technique is that both of the photon energies that are sent through the sample are exponentially attenuated, so that the narrow beam geometry condition is valid. The equations below are then valid [17]

$$N_1 = N_{0,1}\exp(-\mu_1 X) \quad (1)$$

$$N_2 = N_{0,2}\exp(-\mu_2 X) \quad (2)$$

where N_i is the observed transmitted countrate for the energy I, $N_{0,i}$ is the countrate for energy I with no attenuator, μ_i is the linear attenuation coefficient for energy I and X the thickness of the attenuator.

This could also be written as

$$\ln(N_{0,1}/N_1) = \mu_1 X \quad (3)$$

$$\ln(N_{0,2}/N_2) = \mu_2 X \quad (4)$$

The countrate without the attenuator (in this case sample of wood chips) can be determined beside the sample. From Eqs. (3) and (4), the expression of "k" can be calculated as below [17]:

$$k = \left(\ln(N_{0,1}/N_1)\right)/\left(\ln(N_{0,2}/N_2)\right) \quad (5)$$

and because of that Eq. (6) is valid.

$$k = \mu_1/\mu_2 \quad (6)$$

Equation (6) is not dependent of the density and thickness of the sample [17]. The attenuation coefficient (that is linear) of the sample correlates with the atomic number of the sample and the energy that the sample is X-rayed with [18]. It is rather the effective atomic number[1] then the chemical composition of the sample that is affecting the attenuation for each specific compound. The effective atomic number is dependent on how much water the sample consists of, and using Eq. 5 it can be calculated how much water is there in a specific sample, where water is the only variable component [17]. When calculating the effective atomic number for wood, consideration should be taken to the fact that the chemical composition might vary between different trees, even though they are the same species. This might result in a small error when calculating the effective atomic number for wood and in the end affects the accuracy of the MC determination. When the MC varies in the sample, the effective atomic number will vary, and the linear attenuation coefficient will also vary. So by making reference measurements of a material with different MCs, a calibration model for a specific material

[1] The effective atomic number is the calculated average atomic number for a compound or mixture of a material, in this case wood.

can be created. Every measurement is then compared with the reference measurements and the MC in the particular sample can be calculated.

Materials and methods

To test if the calibration model used by Hultnäs et al. [12] could be used to determine the MC in pine, wood chips were collected from a sawmill (Table 1 series A–G). To obtain wood chips with different MCs, the wood chips were spread out on a flat surface indoors and were dried for varying amounts of time to achieve different levels of MC. The chips were mixed once a day so that they would dry evenly [16]. A total of 70 samples were measured ten times each with the MDS. To get a reference value to the results achieved by the MDS, the MC was also determined using the gravimetric method, where the samples were weighed, dried at 103 ± 2 °C until they had reached a stable weight, then the MC could be calculated with formula (7) following the Swedish Standard SS 187170 [19]

$$\frac{\text{weight of wet sample} - \text{weight dry sample}}{\text{weight dry sample}}. \quad (7)$$

When a new calibration model for Scots pine was developed, a new set of pine wood chips were collected (Table 1 series H–L). The same procedure as above was used to obtain chips with different MC levels. Each sample was measured six times using the MDS and the data were then compared to the reference MC, from the gravimetric method described above.

A calibration model based on spruce, pine and different mixtures thereof was applied to the MDS to determine if it was possible to use a single calibration model for the two species. This calibration model was tested using 29 wood chip samples collected from a sawmill, with different mixtures of species and MC (Table 1 samples M–P). To reach a broad range of MC values in the wood chips, the chips underwent a drying treatment that consisted of drying the samples in an oven for a limited amount of time and then allowing them to cool and homogenize in open air for 12 h. When the samples reached the desired MC, they were measured six times using the MDS. Once again, the reference values for the MC values were obtained using the gravimetric technique.

In the last part of the study, 20 samples of spruce wood chips used for pulp production were measured, first frozen and then unfrozen, with the MDS. The samples were stored in a freezer for at least 24 h and to hold a temperature of approximately −18°C. Subsequently, they were each measured four times with the MDS and then were allowed to reach room temperature indoors. The samples were stored in a sample holder equipped with a cover to minimize the loss of moisture due to evaporation. When they reached the desired temperature, each sample was measured again four times.

A linear regression was used to compare the results from the MDS and the gravimetric method. To determine if there was a significant difference between measurements on the frozen and unfrozen samples, a paired two-tailed *t* test was performed.

To calculate a standard deviation (SD) for repeated measurements on the same sample, the following formula was used:

$$\sigma = \sqrt{\frac{\sum((n_i - 1) \times \sigma_i^2)}{\sum(n_i - 1)}} \quad (8)$$

where n_i is the number of measurements per sample performed using the MDS, i sample number, σ_i^2 variance of the measurements per sample.

Table 1 The mixtures of wood chips and the MC of the different samples

Series	Number of samples	Tree species	Mixture (weight %)	MC (%)
A	10	Pine	100	55.4
B	10	Pine	100	53.2
C	10	Pine	100	41.0
D	10	Pine	100	37.8
E	10	Pine	100	28.1
F	10	Pine	100	18.6
G	10	Pine	100	9.4
H	2	Pine	100	54.8
I	2	Pine	100	44.7
J	2	Pine	100	29.9
K	2	Pine	100	21.1
L	4	Pine	100	8.1
M	6	Pine and spruce	30–70	11.5–58.7
N	7	Pine and spruce	50–50	8.0–56.8
O	9	Pine	100	8.8–55.6
P	7	Spruce	100	13.2–58.6

A–G: samples are the pine wood chips used for the MC determination using the calibration model developed for spruce by Hultnäs et al. [12]. H–L: samples used when the MC was determined with a calibration model developed for pine. M–P: samples used, when the MC was determined with a calibration model developed for both species. The MC is the MC from the gravimetric method and expressed in wet basis

Results

The results from the measurements on pine wood chips using the calibration model for spruce are shown in Fig. 2 (calibration line). It is worth noting that the deviation from a perfect correlation line becomes bigger when the samples

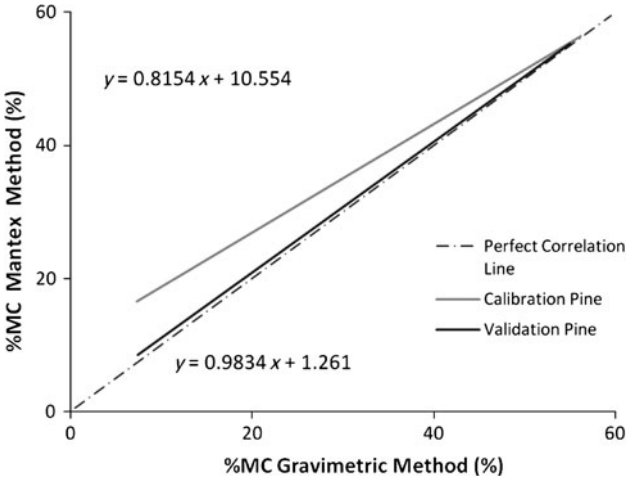

Fig. 2 Comparison of the calibration line (spruce calibration model), and validation line (calibration model developed only for pine). A perfect correlation line is also shown in the diagram. Moisture content is expressed in wet basis

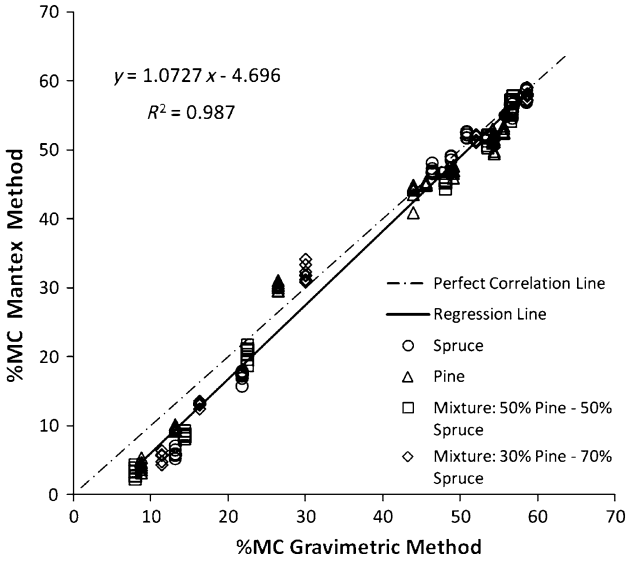

Fig. 3 Correlation between the MDS and the gravimetric method for measuring different mixtures of wood chips using a calibration model developed for both pine and spruce. Moisture content is expressed in wet basis

are getting dryer. The average SD for repeatable measurements on the same sample is 0.45 percentage units. The standard error of estimate (SEE), a measure of the accuracy, was found to be 2.01 %, with an $R^2 = 0.98$.

The result of the regression analysis when using a calibration model for pine is shown in Fig. 2 as the validation line. The SD was again 0.45 percentage units. The SEE decreases to 1.81 % and R^2 was 0.99.

A calibration model, which could be used to measure wood chips of both pine, spruce and different mixtures thereof, where then applied. The results are shown in Fig. 3.

Table 2 provides information about the SD, SEE and the R^2 for each mixture of wood chips. The results are slightly greater variation when compared to previous findings that employed a calibration model for each tree species, with the exception of the R^2 value which is still at the same level.

The results from measurements on frozen and unfrozen samples are presented in Table 3. The greatest deviation in the MC between a frozen and an unfrozen sample was 2.14 percentage units, and the lowest deviation was 0.00 percentage units. For 17 out of the 20 samples, the difference in the results was less than 1 percentage unit. The absolute mean difference between the measurements on all frozen and unfrozen samples was 0.58 percentage units. A t test was performed for frozen and non frozen samples and no significant differences were found between them (0.024 with the limit 2.093) at 95 % confidence levels.

Table 2 Statistics from the measurements of pine and spruce and different mixtures thereof using a calibration model developed to handle both pine and spruce species

Material	SD (%)	SEE (%)	R^2
Pine	0.80	2.57	0.98
Spruce	0.73	1.51	0.99
Mix pine/spruce, 50/50	0.77	1.39	0.99
Mix pine/spruce, 30/70	0.69	2.30	0.99
Total	0.75	2.24	0.99

Moisture content is expressed in wet basis

Table 3 Statistics of the measurements on the same sample that was initially frozen and then unfrozen

	SD (%)	SEE (%)	R^2
Frozen	0.51	1.46	0.99
Not frozen	0.66	1.61	0.99
Total	0.59	0.91	0.99

Discussion

First, we aimed to determine if it was possible to use the calibration model developed for spruce when measuring pine wood chips. The results showed that there were some discrepancies between the results from the MDS and the gravimetric method that was used as a reference. The accuracy, expressed as SEE, decreased from 1.81 % [12] in the spruce study to 2.01 %. The fact that the accuracy in determining the MC of pine using a calibration model developed for spruce was not as good as that of spruce could be explained by the fact that the chemical composition of the two species is slightly different [14], which

results in a different effective atomic number [16]. As shown in Fig. 2, the absolute error increases as the MC decreases; this result indicates that when the sample contains a greater amount of dry matter, the difference in effective atomic number that comes with the differences in the chemical composition, becomes more profound in comparison to the sample containing water, which does not have any variation in its chemical composition nor in its effective atomic number. However, the precision, measured as the SD, was at a very low level of 0.45 %, even lower than in the study performed on spruce (0.54 %) [12]. This is likely due to the use of wood chips from sawmills, while in the spruce study the wood chips came from roundwood. The sawmill chips are more homogenous in their chemical composition due to the fact that they mostly lack heartwood because heartwood typically goes into the sawn goods.

Upon switching to a pine calibration model, the accuracy (SEE) improved and reached the same level as in the spruce study, namely 1.81 % [12]. The precision (SD) remained at the same low level of 0.45 percentage units. The improved precision for pine compared to the spruce study can once again be explained by the origin of the wood chips as discussed above. The increase in accuracy, just underlines the impact of the chemical differences between pine and spruce and the importance of using a specific calibration model if the goal is to keep the accuracy as low as possible. In many situations, the wood chips consist of a mixture of both pine and spruce, and the composition of the mixtures is often unknown. It is therefore desirable to have a single calibration model that is capable of handling mixtures of species. We tested if it was possible to use only one calibration model developed for this purpose and the results demonstrate that both the precision and accuracy were slightly negatively impacted with an SD value of 0.75 % and a SEE value of 2.24 %, in comparison to the previous results. The decrease in accuracy and precision could be expected, as the material that is measured is now more heterogeneous than before. However, as shown in Fig. 3, the MC span between 40 and 60 %, which is a typical range of MC in wood chips for pulp production; the regression line tends to be closer to the perfect correlation line. This result is consistent with the earlier results that demonstrate that drier materials show a higher deviation between the values obtained from the MDS and the gravimetric measurement. The precision of the measurements appears to be slightly decreased when measuring pure pine, or mixture of wood with a high amount of pine in it. The regression line for the mixture of species is below the perfect correlation line (Fig. 2), while for individual species analyzed with a separate calibration model, the data are located above the perfect correlation line. We do not have an explanation for this.

The measurements performed on frozen and unfrozen samples demonstrated that no divergence was observed between the MC values obtained for the frozen and unfrozen material with a 95 % confidence level. Due to the long winters in the Nordic region, it is a prerequisite for any technique that it can handle frozen material. The gravimetric method was used as reference method in this study. This method is standardized and widely accepted, both at research institutes and in industry around the world. However, there are some questions to be raised concerning this method. Errors might occur due to water that is strongly bound to the cell wall still remains upon drying, or that volatile compounds are evaporated during drying. It has also been shown by Björklund and Fryk [20] that the temperature inside the drying ovens could vary significantly from the set value. It is likely that the gravimetric method holds some errors, and it is important to keep this in mind when interpreting different parameters, such as the SEE, because this includes the error from both the MDS and the gravimetric method. Because the gravimetric method destroys the sample, it is also impossible to make repeatable measurements on the same sample and obtain a fair reference value of the precision for the method itself, similar to what has been reported by the MDS. However, since the gravimetric method is a standardized method, commonly accepted and widely used, it is natural to use it as a reference method to a new technique performing the same measurement, so is done in studies by, for example, Duarte Da Paz [7] and Berg et al. [11].

If the level of compensation for pulpwood (in Sweden) is determined by the dry weight of the fibers, the accuracy of the determination needs to be within limits set by the Swedish Forest Agency. Their requirement for loads of wood chips below 50 tons is a deviation of not greater than 9 % from the true value, and for loads above 50 tons, not greater than 6 % [21]. As shown in the results presented in this study, the MDS can provide MC measurements that are well within these limits. The accuracy and precision of the results are partly dependent on which calibration model is applied, where a calibration model developed for a single tree species gives better results than a calibration model developed to handle both pine and spruce. A suggestion for further research could be to test the equipment in more realistic environment, e.g., at a pulp mill, where the technique could be used simultaneously with the gravimetric method to test the technique on a larger quantity.

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